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SUMMARY

Tensile tests using 0.5, 5.0 and 10.0 cm lengths of carbon fibre after heat-treatments to 1000, 1500, 2500 and 3000°C have shown that the length tested has a significant effect on the strengths obtained. With fibres heat-treated to 2500°C, the average strength of 0.5 cm lengths was 400 x 10³ lb in⁻², this being about 30% higher than the corresponding result obtained with 5 cm lengths. The highest strengths were found after the heat-treatment to 1500°C, the average for the 0.5 cm lengths being 450 x 10³ lb in⁻², again about 30% higher than the value for 5 cm lengths. Similar gauge length effects were found after each heat-treatment temperature, and the rathos of the standard deviations of the strengths to the average strengths in general remained constant at about 25%. It appears therefore that the type of defects limiting the strengths and their distribution have not been influenced by the different heat treatments. The strengths of the 0.5 cm lengths have been used to predict values for the longer lengths by means of weak link theory and these values have been compared with the experimental results.

INTRODUCTION

A method has been developed in the Materials Department, R.A.E., of producing carbon fibres of high strength and high modulus, average strengths of about 300 × 10³ lb in⁻² and average Young's moduli of about 60 × 10⁶ lb in⁻² having been obtained¹. Details have since been given of the effect of heat-treatment on the mechanical properties² and on the structure of the fibres³. There is a considerable amount of scatter in the tensile strengths of individual fibres, the standard deviation usually being about 20-30% of the average value. It would be advantageous if this scatter could be reduced and the average strength increased, and as a part of an effort to investigate the nature of the defects limiting the strengths, the effect of gauge length on the tensile strength was investigated. The aim was to establish first of all whether or not there was a gauge length effect and, if so, to find out how the temperature of heat-treatment influenced this effect. Strength measurements on short lengths are also of interest to those studying the mechanical properties of carbon fibre-plastic composites.

2 EXPERIMENTAL

2.1 Preparation of fibres

A batch of proprietary polyacrylonitrile fibre, $1\frac{1}{2}$ denier, 700 filaments/tow, (Ref. No. CF/65) was converted to carbon fibre using the normal R.A.E. process. After an oxidation pre-treatment, the fibres were carbonized in hydrogen up to 1000° C and parts of the batch were later heat-treated in argon for $\frac{1}{2}$ hr at 1500, 2500 and 3000°C respectively, using a 24 in long resistance-heated graphite tube furnace. Temperatures in this furnace were measured by an optical pyrometer.

2.2 Measurement of tensile strengths

Tensile tests on individual fibres were done using 0.5, 5.0 and 10.0 cm gauge lengths, 50 fibres with not more than five taken from any one bundle, being tested in each case with an Instron tensile testing machine. All tests were done using a rate of strain of 1% minute. Prior to testing, the fibres were glued with Durofix across slots of the appropriate length cut in white cards. Their diameters were measured to ±0.1 micron with a microscope fitted with a Watson image-shearing eye-piece. This measuring system had previously been calibrated by means of tungsten wires of known diameters.

After this series of tests some additional testing was done taking five 0.5 cm lengths from each of ten fibres, to see if there was any tendency for fibres to have consistent strengths along their lengths. Some tests were also done in a liquid to see if any failures occurred at the glue line. The liquid, either water or paraffin oil (for the 0.5 cm lengths), was necessary to prevent secondary breaks following the initial fracture. It was held in a beaker resting on the Instron crosshead, the load being applied to the lower grip by means of the double L shaped metal fitting shown in Fig. 1.

3 RESULTS

Table 1 and Fig. 2 show that the length of fibre tested has a significant effect on the strengths obtained, the shorter lengths tending to have higher strengths. For example with fibres heat-treated to 2500°C, the 5 cm lengths had an average strength 13% higher than the 10 cm lengths and the 0.5 cm lengths were higher by 50%. Histograms of the strengths of these fibres are given in Fig. 3. The highest strengths were found after the heat-treatment to 1500°C, as had been expected from previous work² on the effect of heat-treatment temperature on mechanical properties.

The strength distributions of the individual results obtained with 0.5 and 5.0 cm lengths are shown in Figs. 4-7. These figures which show how the fraction of fibres unbroken varies with the applied stress, can be used to predict the strength distributions of other lengths and will be discussed in more detail in the next section.

Table 2 gives the results obtained by testing several different 0.5 cm lengths from another ten fibres of the batch heat-treated to 2500°C. It can be seen that, although most fibres had average strengths not much different from the overall average, No. 3 was consistently higher and No. 7 consistently lower in strength.

4 DISCUSSION

The results show clearly that as the fibre gauge length decreases the observed average tensile strength increases. This effect could be due to failures occurring at flaws scattered along the lengths of the fibres; it is more likely that a severe flaw will be present the longer the gauge length. Gauge length - strength effects have also been found with fibres of other types such as steel wires⁵, textile fibres⁶ and glass fibres⁷.

A weak link theory^{8,9} has been developed to relate the strengths of different lengths; it assumes that long lengths are made up of random

selections of short lengths or links and that the strengths of these links have a normal distribution. Tippett has shown how the average strengths and the standard deviations of chains of n links can be expressed in terms of the strengths of the individual links. Table 3 taken from a paper by Tucker gives Tippett's relations for several values of n the number of links. This method has been used here, the 0.5 cm lengths being regarded as the links and used to predict the average values that would be expected for the 5 and 10 cm lengths. These predicted values, together with the corresponding experimental results, are given in Table 4. It can be seen that, although the figures agree fairly well, the predicted strengths are consistently lower than the experimental results, the differences ranging between 6-17%

The strength distributions for the 0.5 cm lengths, shown in Figs.4-7, have also been used to predict results for 5 cm lengths. These figures show the fraction of fibres unbroken at any given applied stress and this can be regarded as the probability that a fibre will be able to withstand this stress. Therefore if the probability of selecting a 0.5 cm length that will withstand a given stress is p, and the 5 cm lengths are regarded as being made up of ten 0.5 cm lengths selected at random, then the probability of obtaining a 5 cm length that will not break at the same stress is $p^{5.0/0.5}$, i.e. p^{10} . In this way predicted strength distributions were plotted for 5 cm lengths and again it can be seen that the predicted strengths are somewhat lower than the experimental values.

These discrepancies between predicted and experimental values could be due to either or both of the following:-

- (a) A tendency for fibres to have consistent strengths along their lengths, i.e. for individual fibres to be strong or weak independent of gauge length.
- (b) Some of the measured strengths of 0.5 cm lengths being lower than the true strengths, due to premature failures at the point where the fibre is glued to the mounting card. This could occur because of a lack of alignment during testing.

Therefore to find out if fibres did tend to be of uniform strength some further tests were done on fibres heat-treated at 2500°C. Five 0.5 cm lengths were tested from each of ten fibres taken from two bundles, five lengths being as many as could easily be mounted from the 15 cm lengths of

fibre. On some occasions only three or four lengths were tested due to the difficulties in handling these short lengths. The results are given in Table 2. The overall average for this series of tests was 435×10^3 lb in $^{-2}$ with a standard deviation of 105×10^3 lb in⁻², the corresponding figures for the first series given in Table 1 being 400×10^3 lb in $^{-2}$ and 93×10^3 lb in $^{-2}$ respectively. As this difference in the averages, 35×10^3 lb in $^{-2}$, had a standard error of about 20 × 10³ lb in -2 it is possibly significant at the 10% level and it suggests that there was some variation in the average strengths of fibres in different bundles. As mentioned in Section 2.2, the occurrence of this type of sampling error in the normal tests was minimized by not taking more than five fibres from any one bundle, the bundles being selected from the batches at random. Although the results for most of the ten fibres had a considerable scatter, fibre No. 3 seemed to be consistently high and fibre No. 7 consistently low in strength. No. 3, for example, had an average strength that was 130×10^3 lb in⁻² above the average for the first series. There seems to be some tendency therefore for fibres to be of uniform strength which has contributed to some extent to the discrepancies between predicted and experimental strengths for the 5 and 10 cm lengths.

Some fibres were then tested immersed in a liquid in an effort to find out whether fractures were tending to occur at the glue line. With 5 cm lengths water was used to prevent secondary breaks and it was found that tests could be done satisfactorily, i.e. without failures at the glue line, when the fibres were aligned with an accuracy comparable to that achieved in the normal tests. When using 0.5 cm lengths a more viscous liquid, paraffin oil, was necessary to prevent secondary fractures. Ten lengths were tested and of these three broke at the glue line, suggesting that this type of premature failure could also have occurred in the normal tests, although probably not in as many as 30% of them as it was easier to align the short lengths of fibre when testing in air. The strength histogram for the 0.5 cm lengths shown in Fig.3 does not show an unusual number of low results as might have been expected if many of the fibres had failed prematurely at the ends.

An important feature of the results, as shown in Fig.2, is that the variations of strength with length were similar after the different heat-treatments. This, taken in conjunction with the fact that the standard deviations when expressed as percentages of the average strengths, as in Table 1, were approximately constant for each group of tests, suggests that

although the heat-treatments have influenced the strengths the type of defects and their distribution have not been altered. It would be interesting to examine the effect of gauge length on strength of fibres heated to temperatures between 220-1000°C as extensive chemical changes take place in this region and little is known about the effect of these changes on the mechanical properties. Some work at present in progress on the tensile testing of precursor fibres in liquid nitrogen¹⁰ could also be extended by using different gauge lengths. This would be worthwhile as it might show whether the defects in carbon fibres can be linked with defects in the original starting fibre.

5 <u>CONCLUSIONS</u>

- (1) It has been shown that the length of carbon fibre tested does have a significant effect on the tensile strength obtained. For example, with fibres heat-treated to 2500° C, the average strength of 0.5 cm lengths was 400×10^{3} lb in⁻² this being about 30% higher than the result obtained with 5 cm lengths.
- (2) The highest strengths were found after the heat-treatment at 1500° C, the average for the 0.5 cm lengths being 450×10^{3} lb in 2, again about 30% higher than the corresponding value for the 5 cm lengths.
- (3) The type of defects and their distribution in the fibres does not appear to have been influenced by heat-treatment temperature, as similar gauge length effects were found after each heat-treatment and the standard deviations expressed as percentages of the averages in general remained constant at about 25%.
- (4) The results were correlated by means of a weak link theory that assumes that longer lengths consist of random selections of short lengths or links. Fair agreement was found although the predicted values for the 5 and 10 cm lengths were lower than the experimental results by 6-17%. The discrepancies could be partly due to a slight tendency for some fibres to be consistently strong or weak along their lengths and possibly also due to some premature end failures in the 0.5 cm tests because of the difficulty of aligning these lengths satisfactorily.

Acknowledgements

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Table 1

EFFECT OF GAUGE LENGTH AND TEMPERATURE OF HEAT TREATMENT ON THE

AVERAGE TENSILE STRENGTHS OF CARBON FIBRES

Temperature of heat treatment (°C)	Gauge length (cm)	Average diameter (microns)	Average tensile strength (lb in ⁻²)		Standard deviation Average strength (%)
1000	0•5 5•0 10•0	7•0 7•1 7•4	291×10^3 213×10^3 180×10^3	53 × 10 ³	22 25 22
1500	0•5 5•0 10•0	6.8 6.9 7.0	450 × 10 ³ 346 × 10 ³ 309 × 10 ³	72 × 10 ³	22 21 23
2500	0•5 5•0 10•0	6•4 6•5 6•6	400×10^{3} 300×10^{3} 266×10^{3}	84×10^{3}	23 28 21
3000	0•5 5•0 10•0	6.1 6.2 6.0	330 × 10 ³ 213 × 10 ³ 205 × 10 ³		26 33 38

STRENGTHS OF DIFFERENT 0.5 cm LENGTHS TAKEN FROM FIBRES

HEAT-TREATED TO 2500°C

Fibre	No•	Individual strengths of 0.5 cm lengths (lb in-2)	Average strength of 0.5 cm lengths from each fibre (lb in 2)	
1		510 × 10 ³ 384 " " 445 " " 460 " " 517 " "	463 × 10 ³	
2		502 × 10 ³ 532 " " 341 " " 470 " " 314 " "	432 × 10 ³	
3		548 × 103 437 " " 501 " " 582 " " 582 " "	530 × 10 ³	
4		516 × 10 ³ 337 " " 509 " " 544 " " 372 " "	456 × 10 ³	435 × 10 ³ (standard dev <u>j</u> ation)
5		268 × 10 ³ 203 " " 667 " " 492 " " 224	371 × 10 ³	105 × 10 ³
6		549 × 10 ³ 491 " " 412 " " 497 " " 443 " "	478 × 10 ³	
7		338 × 10 ³ 356 " " 294 " " 265 " " 383 " "	345 × 10 ³	
8		292 × 10 ³ 452 " " 579 " " 477 " "	450 × 10 ³	

Table 2 (Contd)

Fibre No.	Individual strengths of 0.5 cm lengths (lb in-2)	Average strength of 0.5 cm lengths from each fibre (lb in 2)	Overall average strength of the O.5 cm lengths (lb in ⁻²)
9	387 × 10 ³ 376 " " 458 " "	407 × 10 ³	
10	545 × 10 3 43 3 " " 278 " "	419 × 10 ³	

Table 3

THE AVERAGE STRENGTHS AND THE STANDARD DEVIATIONS OF STRENGTHS

FOR CHAINS OF n LINKS^{8,9}.

Number of links, n	Average strength of chains	Standard deviation of chains
· 1	М	σ
2	м - 0.56 о	0 . 83 σ
5	м - 1•16 о	0 . 67 σ
10	M - 1.54 σ	0. 59 σ
20	м - 1.87 б	0 •5 3 σ
100	М - 2.51 о	0 . 43 σ

Table 4

COMPARISON OF STRENGTHS PREDICTED BY WEAK LINK THEORY USING
TIPPETT'S METHOD^{8,9} WITH MEASURED VALUES FOR 5 AND 10 cm LENGTHS

Temperature of heat-	Gauge	Average tensile strength (lb in -2)		Standard deviation of strengths (lb in ⁻²)	
	length (cm)	Predicted	Experimental	Predicted	Experimental
1000	5 10	191 × 10 ³ 169 × 10 ³	213 × 10 ³ 180 × 10 ³	38 × 10 ³ 34 × 10 ³	53 × 10 ³ 40 × 10 ³
1500	5 10	300 × 10 ³ 268 × 10 ³	346 × 10 ³ 309 × 10 ³	58 × 10 ³ 52 × 10 ³	72 × 10 ³ 72 × 10 ³
2500	5 10	257 × 10 ³ 226 × 10 ³	300 × 10 ³ 266 × 10 ³	55 × 10 ³ 49 × 10 ³	84 × 10 ³ 55 × 10 ³
3000	5 10	198 × 10 ³ 169 × 10 ³	213 × 10 ³ 205 × 10 ³	51 × 10 ³ 46 × 10 ³	70 × 10 ³ 77 × 10 ³

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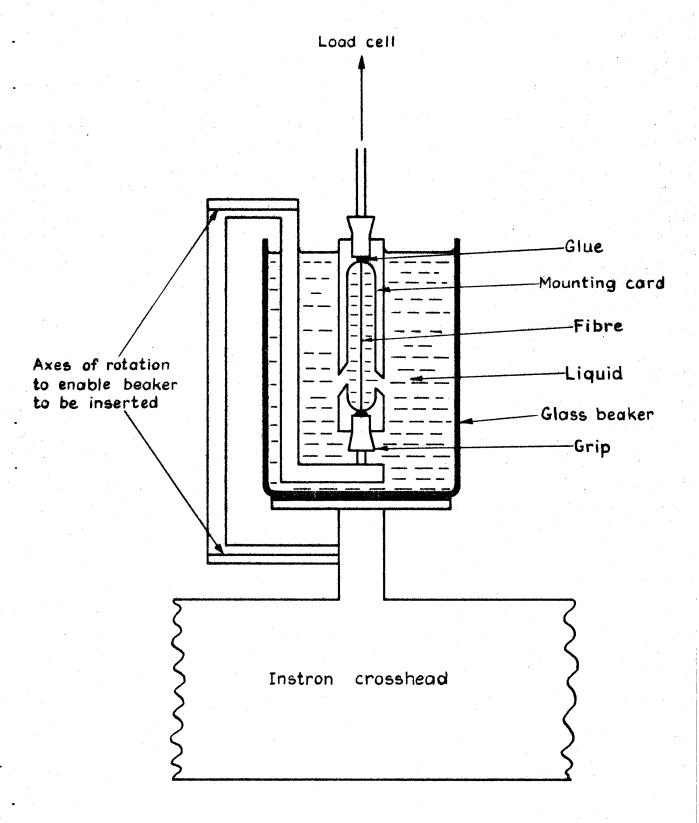


Fig. 1 Arrangement for testing fibres immersed in a liquid

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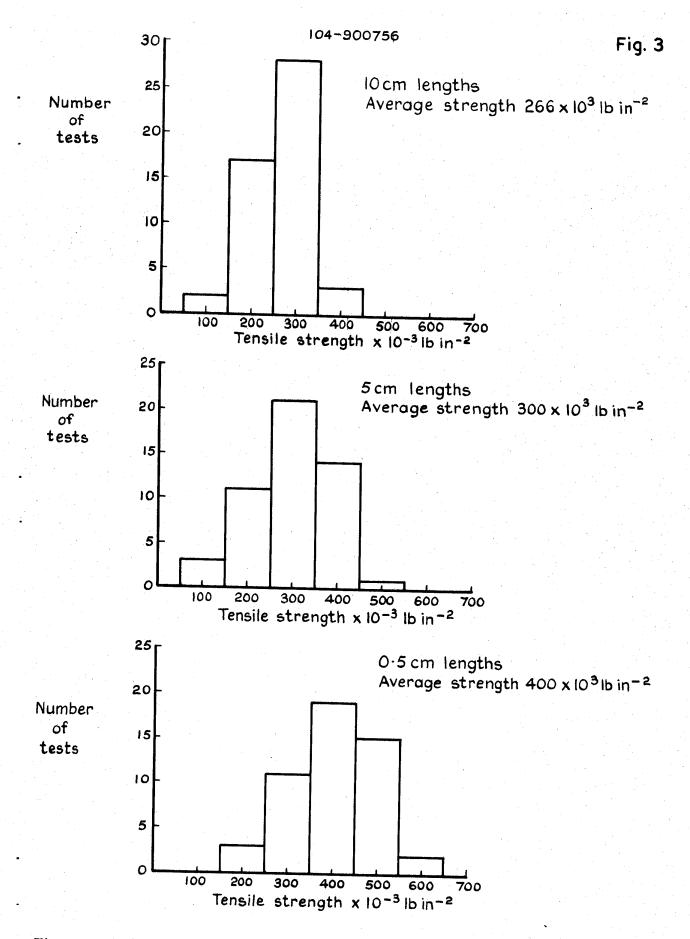


Fig. 3 Histograms of tensile strengths of 0.5, 5.0 and 10.0cm lengths of fibre after the heat-treatment to 2500 °C

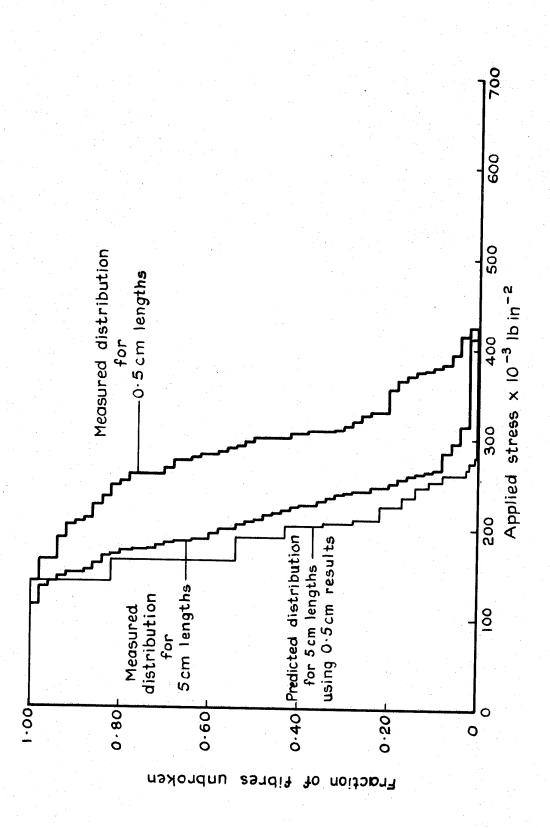


Fig. 4 Strength distributions of fibres after being carbonized to 1000 °C



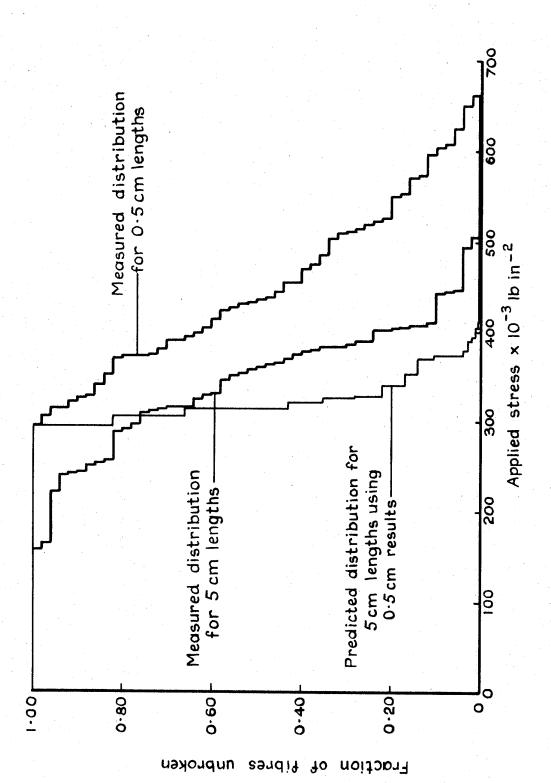
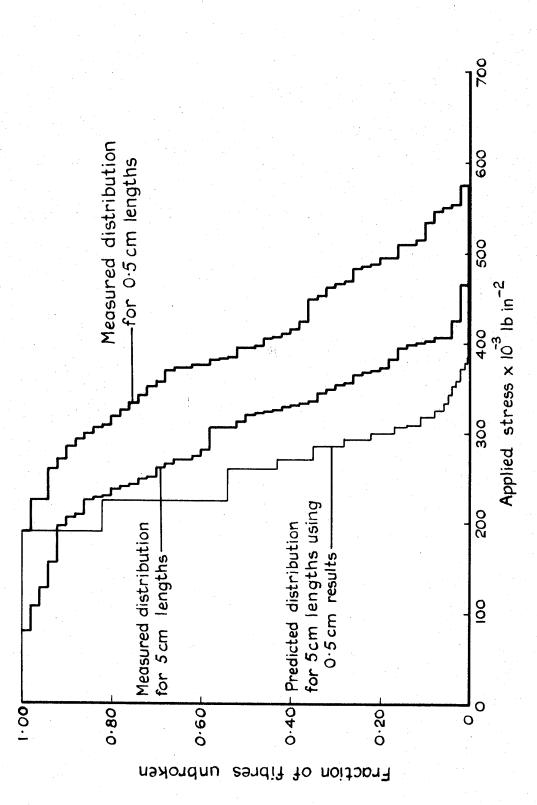


Fig. 5 Strength distributions of fibres after being heat-treated to 1500 °C



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Fig. 6 Strength distributions of fibres after being heat-treated to 2500°C

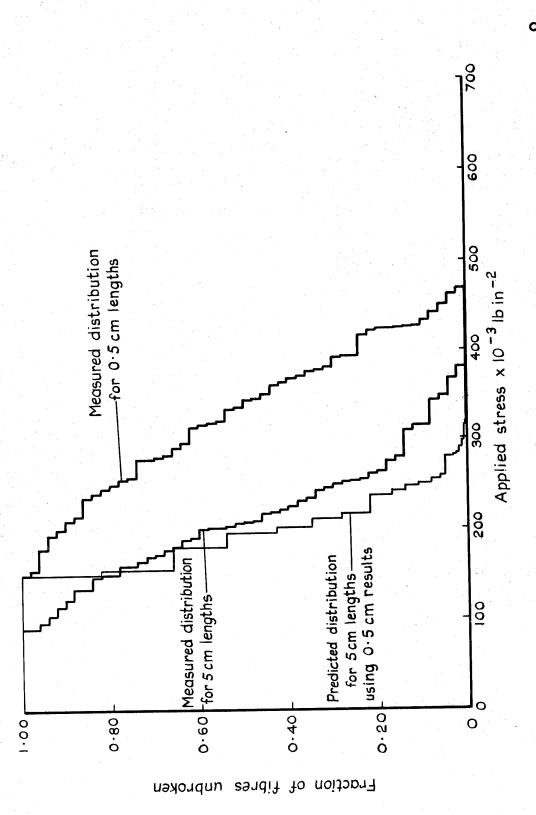


Fig. 7 Strength distributions of fibres after being heat-treated to 3000 °C